

**PRESENCE OF SOIL-APPLIED HERBICIDES
IN THREE RIGHTS-OF-WAY INFILTRATION BASINS
IN SAN JOAQUIN COUNTY, CALIFORNIA**

By

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ABSTRACT

Rights-of-way infiltration basins are natural or excavated depressions in the ground used to collect highway storm water runoff in order to provide adequate roadway drainage. Water collected in the basins is disposed by infiltration into the soil and by evaporation. Storm runoff which has moved across roadsides recently treated with soil-applied herbicides may transport herbicide residues to basins, where there is the potential for herbicide transport to ground water by soil infiltration of accumulated water. This study was conducted to determine the presence of soil-applied herbicide residues in water and soil samples at three Caltrans infiltration basins in San Joaquin County. Water samples were collected from an inflow canal delivering runoff to a basin, from basin storage, and from basin drywells. Soil at each basin was sampled at 1-foot increments. Maximum herbicide concentrations detected in water sampled from the inflow canal were diuron at 1.42 ppb, oryzalin at 27.40 ppb, oxyfluorfen at 5.62 ppb, and simazine at 83.47 ppb. Maximum concentrations detected in water sampled from basin storage were bromacil at 25.62 ppb, diuron at 36.32 ppb, oryzalin at 12.50 ppb, oxyfluorfen at 3.47 ppb, and simazine at 78.80 ppb. In water sampled from basin drywells, maximum concentrations were diuron at 0.46 ppb, oxyfluorfen at 0.28 ppb, and simazine at 17.88 ppb. Simazine and diuron had not been applied to the highway right-of-way in the Peltier basin drainage area and probably originated from agricultural agricultural land. The only herbicide detected in basin soil was simazine at 36.0 ppb. Detection of herbicide residues in water sampled from storm runoff flowing into a basin, and in water sampled from basins and basin drywells, indicates a potential for the herbicides to be transported to ground water by soil infiltration. Detection of herbicides other than those used exclusively for rights-of-way weed control suggests a contribution of herbicide residues from adjacent properties.

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DISCLAIMER

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I. INTRODUCTION

The disposal of highway storm water runoff is a growing problem within densely populated areas of California. Urbanization of rural areas along major freeways and interchange systems has increased runoff and eliminated natural drainage courses. The California Department of Transportation (Caltrans) investigated alternative drainage systems to alleviate the growing surface water disposal problem. The flat terrain and limited natural water channels in some regions required surface runoff to be stored and infiltrated, rather than transported into already overburdened drainage channels. Collection of runoff water in rights-of-way infiltration basins has proved to be a very effective and practical approach (Jackura, 1980).

Soil application of preemergent herbicides during the fall and winter is the most widely used practice for rights-of-way vegetation management in California. The success of preemergent herbicides depends on the amount and timing of rainfall following application. Preemergent herbicides are usually applied soon after the beginning of the fall rainy season. This avoids application to dry, dusty soil surfaces which may be eroded from the site, and allows rainfall to move the herbicide into the upper layers of soil before weed seed germination. Herbicide residues may be washed from the soil surface and carried off-site in storm water runoff, especially when there is a short time interval between herbicide application and excessive or intense rainfall. Runoff may carry

residues to infiltration basins, where there is the potential for transport to ground water by soil infiltration.

Although Caltrans and Departments of Transportation from other states have extensively studied the composition of highway storm water runoff (e.g. Driscoll et al., 1990; Hoffman et al., 1985; Mar et al., 1982; Racin et al., 1982), none of this research has measured concentrations of herbicides in runoff. Origins of potential herbicide movement in runoff to basins often includes more than just the highway rights-of-way in the immediate vicinity; water draining naturally onto the right-of-way from adjoining properties must be accommodated as well. However, adjacent property owners cannot artificially divert water onto the right-of-way, as they can be held liable for adversely altering natural flows (Jackura, 1980).

This study was conducted by the Environmental Monitoring and Pest Management Branch of the Department of Pesticide Regulation (DPR) in cooperation with Caltrans to investigate the presence of herbicide residues in rights-of-way infiltration basins. The objective was to determine the presence of soil-applied herbicide residues in: 1) water flowing into basins, 2) water collected from basins, and 3) soil sampled from basins. The study was conducted from January through May, 1992 in San Joaquin County (Caltrans District 10). The selection of herbicide residues analyzed was based on herbicides applied to the highway right-of-way and to adjacent agricultural land.

II. MATERIALS AND METHODS

STUDY SITES

Site selection

Sites were selected based on drainage patterns, herbicide use in the drainage areas, and on runoff volume; the amount of stored water was to support periodic sampling throughout the rainy season. Three Caltrans infiltration basins in San Joaquin County were selected for study; the Peltier basin near Lodi, and two basins near Manteca (Figure 1).

Caltrans District 10 personnel provided drainage diagrams and informative tours of candidate sites. Pesticide use history in the drainage areas was obtained from Caltrans (Johnson, 1992; Shields, 1992), the San Joaquin County Agricultural Commissioner's office (Helmar, 1992), and the 1991 DPR Pesticide Use Report.

Site description

Peltier basin

The Peltier basin is located in the northeast quadrant of the Highway 99 and Peltier Road interchange north of Lodi in San Joaquin County (Figures 1A, 2). The basin was constructed in 1960 and is a major drainage feature in the area, providing a reservoir for rapid drainage of highway runoff, and storm and irrigation runoff from a 5-square mile agricultural area adjacent to the highway right-of-way (Figure 3). The 1-1/2 acre basin has a storage capacity of approximately 5 acre-feet and stores water continuously from late January to mid-April. Depth of the basin is 5 feet. Storage capacity is limited by the height of the outflow drain, 36 inches above the basin floor. The outflow drain empties into a canal

Figure 1. Study site locations.

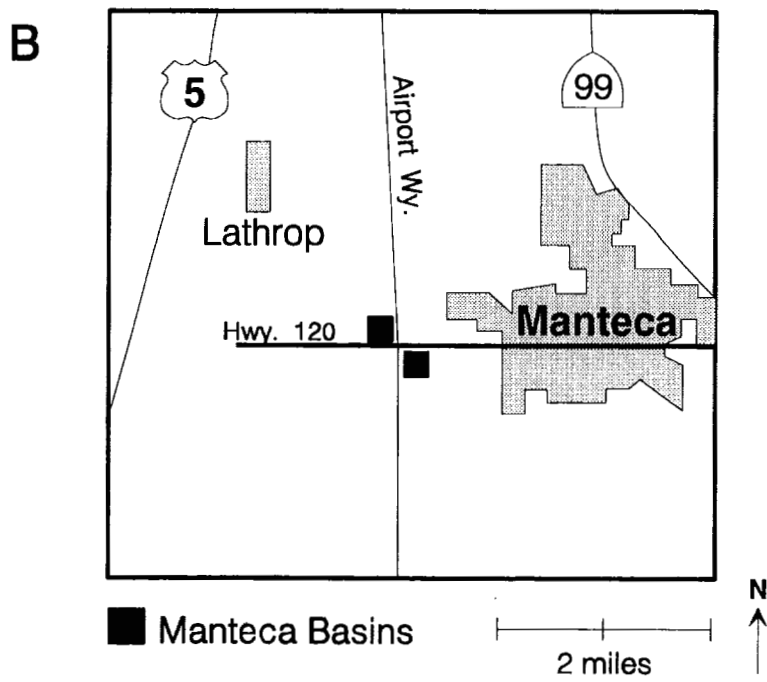
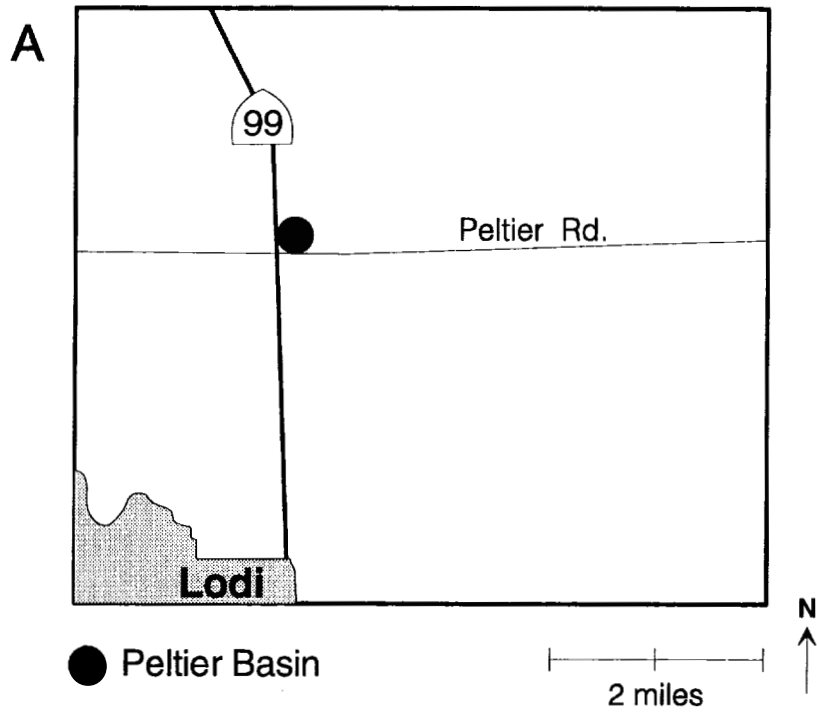


Figure 2. Peltier basin and sampling sites.

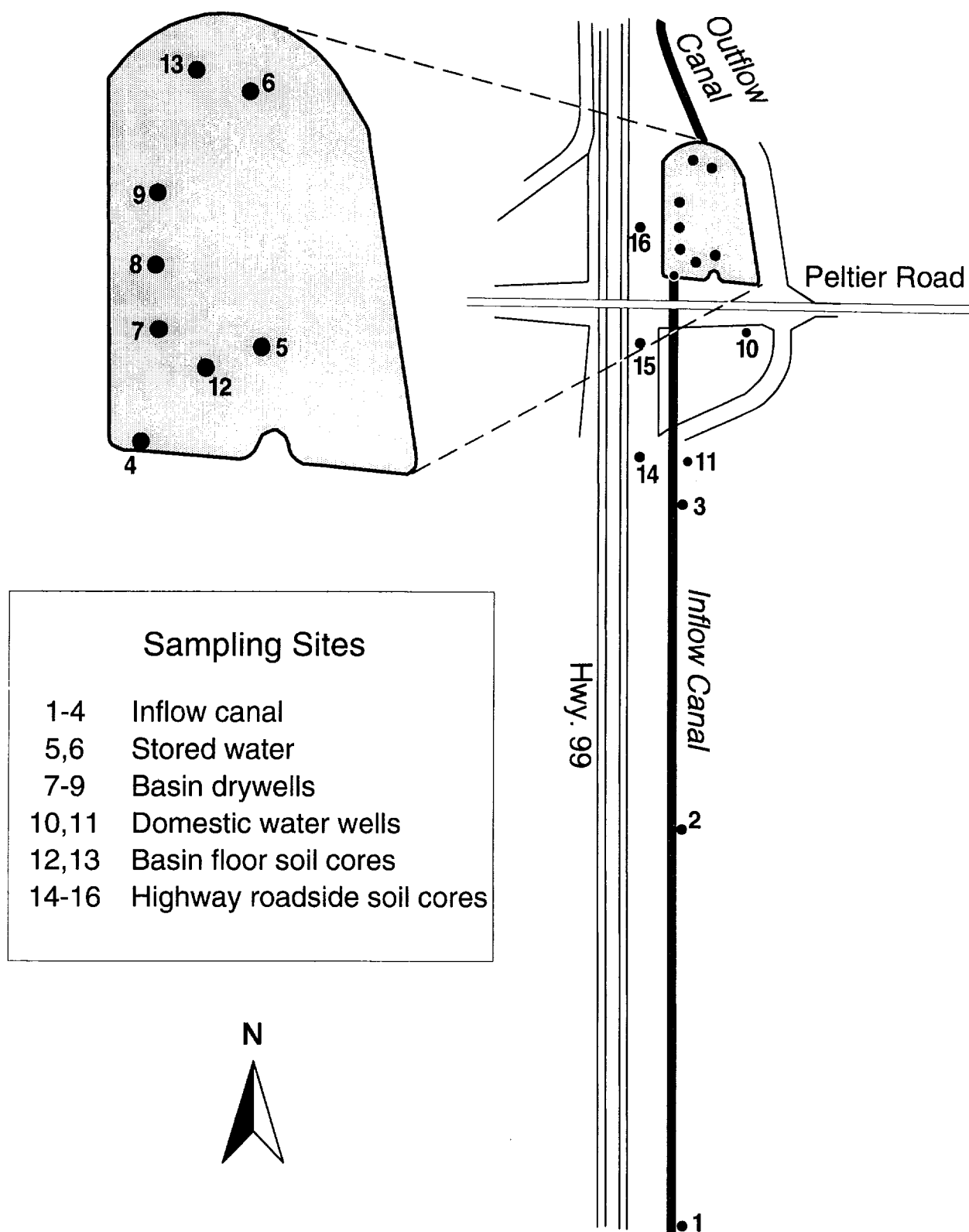
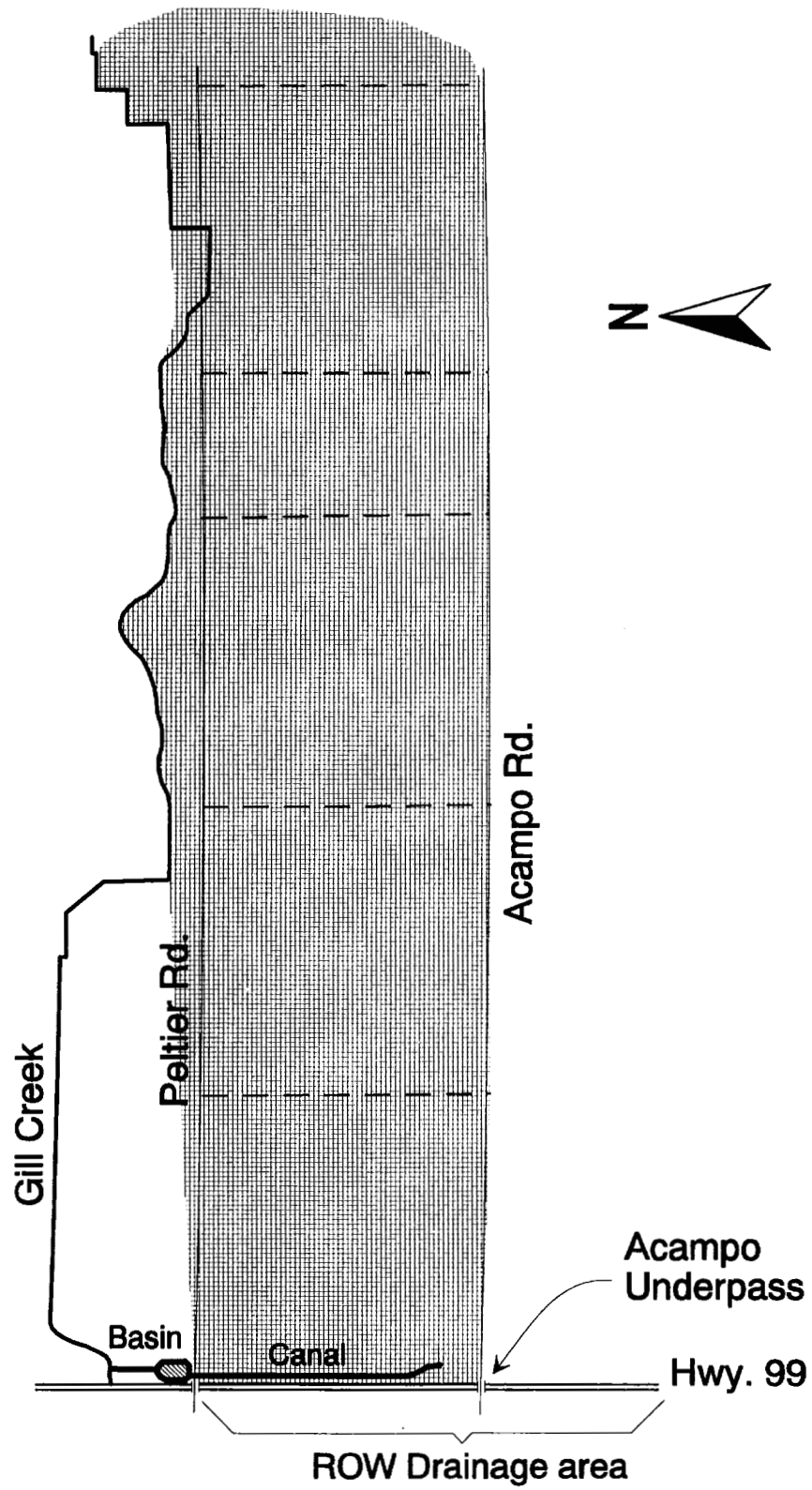


Figure 3. Peltier basin drainage area outside the right-of-way (shaded area).



which conveys water north from the basin, eventually to Gill Creek. Three drywells in the basin, each 35 feet deep, provide faster, higher-volume infiltration (Figure 4). Depth from surface to ground water was estimated to be 65-70 feet (Figure 5A). The vertical setback distance from the bottom of the drywell to the water table was estimated to be 40 feet. Domestic water wells are located on neighboring properties.

Runoff is delivered to the basin in a concrete-lined inflow canal, extending south of the basin nearly a mile in length (Figure 3). The canal was built primarily for drainage of the highway underpass at Acampo Road, one mile south of the basin. The Caltrans pump station at the underpass pumps water from the underpass into the canal for drainage north to the basin. The canal also conveys direct runoff into the canal from highway and frontage road rights-of-way and adjoining properties. A major change to this basin drainage has been the addition of drainage waters from the county storm drainage system, which collects storm and irrigation runoff from the agricultural area extending east from the right-of-way.

Since 1988, Caltrans has applied single, annual soil applications of oryzalin at 4 lbs. active ingredient (a.i.)/acre and oxyfluorfen at 1.6 lbs. a.i./acre to the Highway 99 roadside and median strips; the 1992 treatment was applied January 21-24 (Shields, 1992)(Table 1). The agricultural land in the drainage area is planted primarily to vineyards, where the soil-applied herbicides of greatest use are simazine, oryzalin, sethoxydim, diuron, and norflurazon (Helmar, 1992).

Figure 4. Peltier basin drywell.

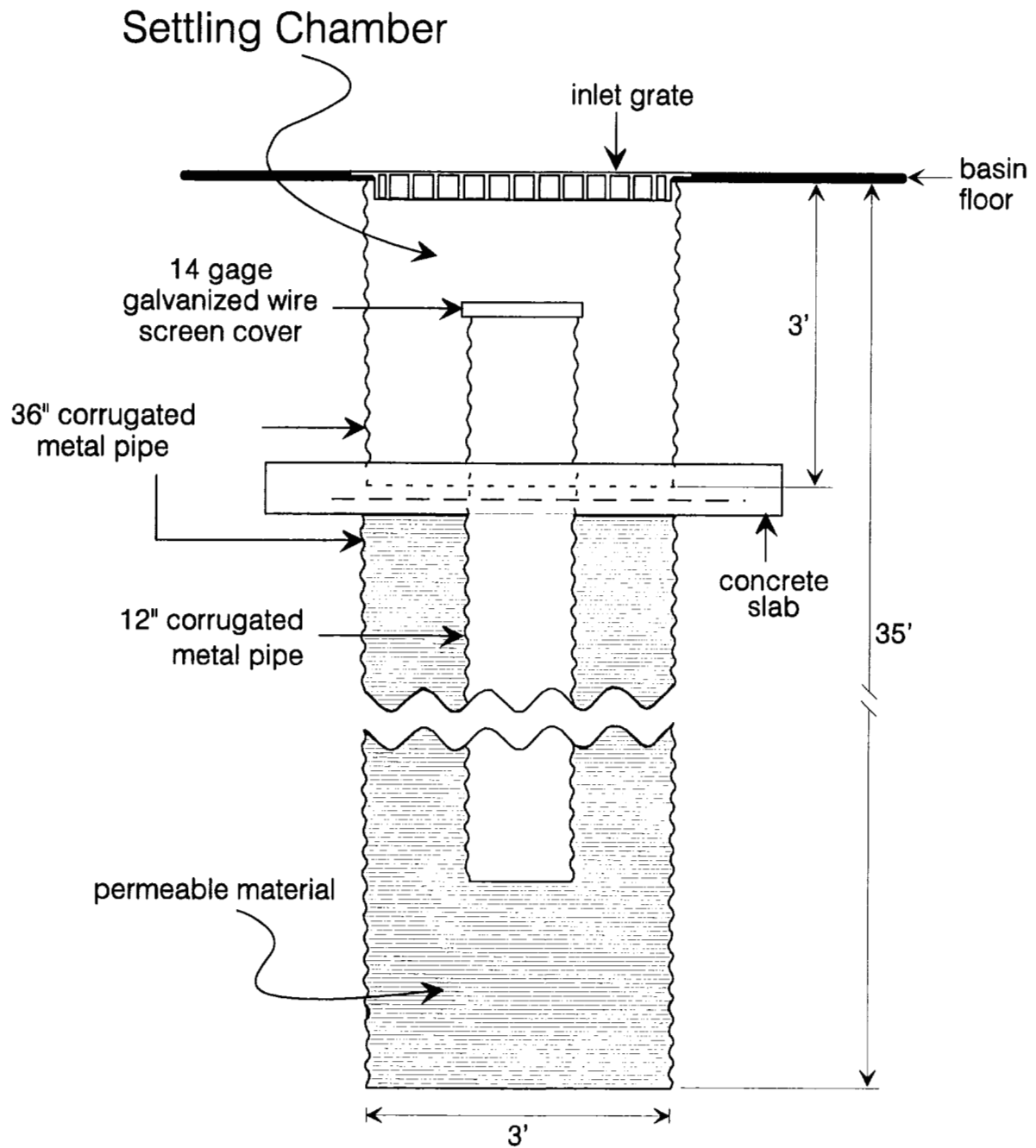
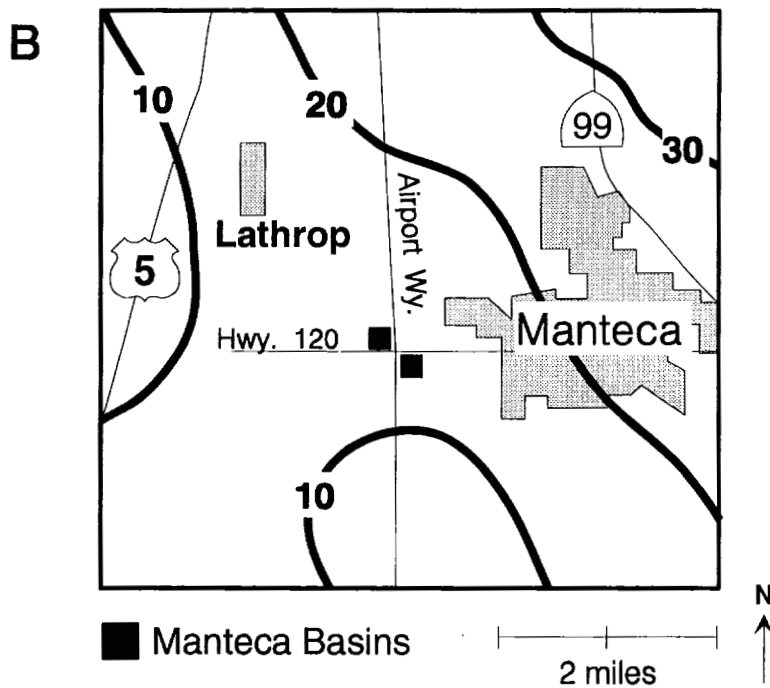
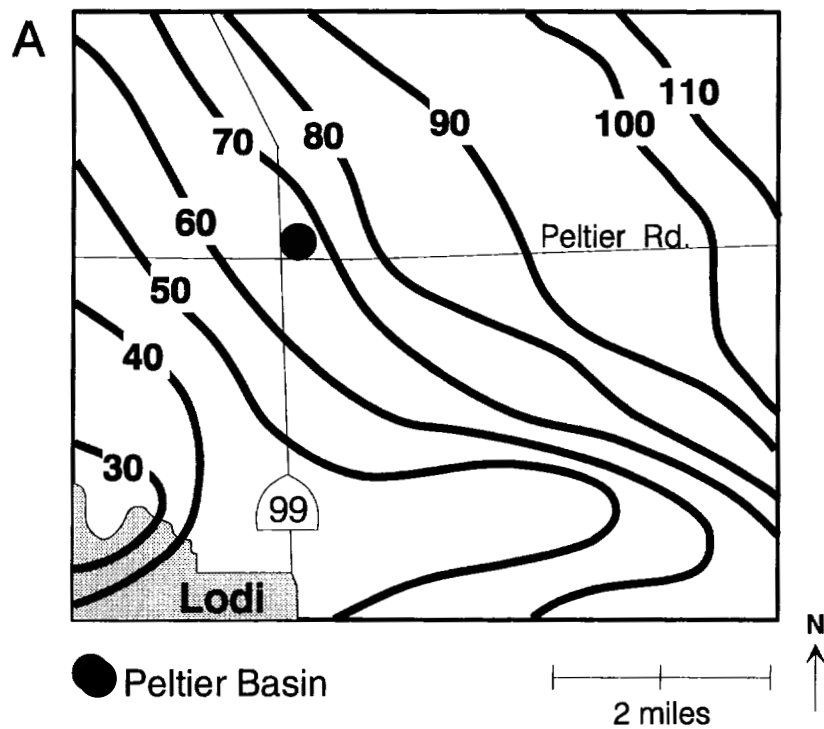


Figure 5. Lines of equal depth to water in wells for the Peltier and Manteca sites.



Source: San Joaquin County Flood Control and Water Conservation District, Spring 1989

Table 1. Herbicide applications along highway roadsides in basin drainage areas.

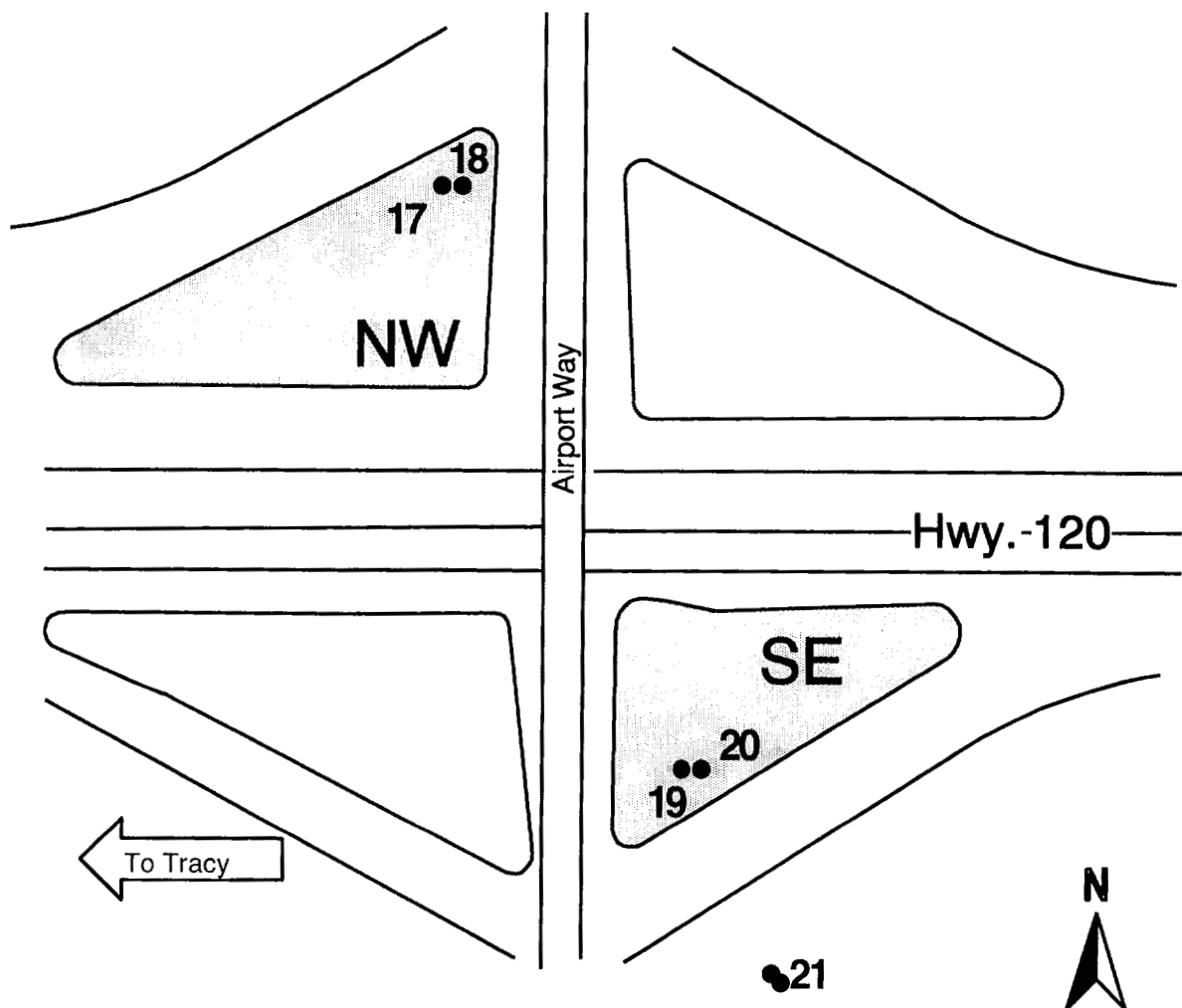
Highway 99 Peltier basin drainage		
<u>Herbicide (a.i.)</u>	<u>Rate (a.i./acre)</u>	<u>Date applied</u>
oryzalin (Caltrans)	4.0 lb	Jan. 21-24, 1992
oxyfluorfen (Caltrans)	1.6 lb	Jan. 21-24, 1992
Highway 120 Manteca basins drainage		
<u>Herbicide (a.i.)</u>	<u>Rate (a.i./acre)</u>	<u>Date applied</u>
bromacil (Caltrans)	2.4 lb	Feb. 12-13, 1992
diuron (Caltrans)	2.4 lb	Feb. 12-13, 1992
simazine (County)	7.2 lb	Dec. 11, 1991

Simazine is the most widely-used soil-applied herbicide in the vineyards; oxyfluorfen is registered only for non-bearing vineyards and it is not extensively used. Also in this area, the San Joaquin County Department of Public Works treats county roadsides with simazine and, to a much lesser extent, with diuron (Helmar, 1992). Other noncrop sites of soil herbicide use in the drainage area include railroad and utility rights-of-way, storage sites, equipment yards, and parking areas.

Manteca basins

The two Manteca basins are located in opposite quadrants of the Highway 120 and Airport Way interchange, one mile southwest of Manteca in San Joaquin County (Figure 1B). These basins will be referred to individually as the 'Manteca SE basin' in the southeast quadrant and 'Manteca NW basin' in the northwest quadrant (Figure 6). The basins were excavated during highway construction in 1980. Each basin is triangular

Figure 6. Manteca basins and sampling sites.



Sampling Sites			
NW basin		SE basin	
17	Stored water	19	Stored water
18	Basin floor soil core	20	Basin floor soil core
		21	Domestic water well

in shape and sloped gradually from the far end of each quadrant to a deepest point near the Airport Way overpass ramp bordering each basin. There are no inflow conveyance structures; all inflow is surface runoff directly into the basins from the Highway 120 roadside, on-ramps, and from the overpass ramp. There is no runoff to the basins from properties outside the right-of-way (Kraetsch, 1992). During the last 6 years of unusually low rainfall, the amount of water stored in the basins has been minimal. During this study, the depth of stored water did not exceed 6 inches in either basin. Depth from surface to ground water was estimated at 10 feet (Figure 5B). Depth from the floor of each basin to ground water was estimated at 5 feet.

Potential sources of herbicide off-site movement to the Manteca basins were the Highway 120 roadside treatment by Caltrans, and the overpass ramp roadside treatment by the County Department of Agriculture (Table 1). Since construction of the highway and basins in 1980, Caltrans has annually applied 2.4 lbs a.i./acre bromacil + 2.4 lbs. a.i./acre diuron (6 lbs./acre treatment rate of Krovar*) to the Highway 120 roadside and on-ramps; the 1992 treatment was applied February 12-13 (Shields, 1992). The County applies a single, annual application of simazine to the Airport Way overpass ramp (diuron was last applied in 1989). Application rates (a.i./acre) for overpass ramp treatment during the last 3 years were: 7.2 lbs. simazine (applied 12/11/91); 7.2 lbs. simazine (applied 12/14/90); 3.2 lbs. simazine + 3.2 lbs. diuron (applied 11/2/89) (Helmar, 1992)(Table 1).

STUDY DESIGN

Water and soil sampling in the drainage areas was initiated after Caltrans roadside treatments. Water samples were collected during rainstorms or the day following. Six sampling visits were conducted at 2-3 week intervals from late-January to mid-April. Sample collection sites are depicted in Figure 2 for the Peltier site and in Figure 6 for the Manteca sites.

Peltier basin

Water samples were collected from the inflow canal, from water stored in the basin, from the three basin drywells, and from two domestic water wells on private properties near the basin. Canal, stored water, and drywell water samples were analyzed for diuron, oryzalin, oxyfluorfen and simazine. Domestic well water samples were analyzed for atrazine, bromacil, diuron, oryzalin, oxyfluorfen, prometon and simazine. Atrazine and prometon were included as these analyses are routinely performed on well samples collected by the DPR, Environmental Hazards Assessment Program for AB 2021 (Ground Water Contamination Prevention Act) well sampling.

Basin soil was sampled to a depth of 10 feet following final drainage of the basin in April. The samples were analyzed for diuron, oxyfluorfen and simazine; no laboratory method was available for oryzalin analysis at the time of this study. Background soil sampling was not conducted because the study was initiated after the beginning of seasonal storm water runoff drainage to the basin.

Soil was sampled beneath the Caltrans treatment strip on the Highway 99 roadside bordering the west side of the basin. The presence of simazine or diuron residues near the surface (remaining from Caltrans treatments prior to 1988) would indicate that previous applications of these herbicides to the roadside may have also been a source of residues.

Manteca basins

Water samples were collected from water stored in the basins, and from one domestic water well on private property near the southern perimeter of the SE basin. Basin water samples were analyzed for bromacil, diuron, and simazine. Domestic water well samples were analyzed for atrazine, bromacil, diuron, prometon and simazine.

Single soil cores were taken from each basin following final seasonal drainage of the basin in April. The shallow water table limited coring depth to 4 feet in the SE basin, and to 2 1/2 feet in the NW basin. Soil samples from both basins were analyzed for bromacil, diuron and simazine. Only the NW basin was sampled for background soil concentrations. A single core was taken from the NW basin before the Caltrans bromacil and diuron treatment, but not before the County simazine treatment; for this reason, the background samples were analyzed only for bromacil and diuron.

SAMPLING METHODS

Water sampling

All water samples were collected in 1-liter amber glass bottles which were immersed below the surface of the water and allowed to fill. When

water was too shallow for immersion, a second clean bottle was used to fill the sample bottle. Samples were transported on wet ice and refrigerated in Sacramento until transfer to the laboratory for chemical analysis.

Samples of water stored in the Peltier basin were collected from the north and south sides of the basin, 15-30 feet from the water's edge. Samples of water stored in the Manteca basins were collected where water was deep enough to submerge sample bottles. Water samples from each drywell settling chamber were collected with hand-operated vacuum pumps and teflon tubing. The tubing was inserted into the chamber through the drywell inlet grate. A 1-liter glass amber sample bottle was filled from the 36-inch deep settling chamber in each of 3 drywells.

Domestic water well sampling methods were used as described by Sava, 1986.

Soil core sampling

A 4-inch inside diameter (ID) hand auger was used for all soil coring. Prior to coring, a PVC collar was hammered into the soil to prevent crumbling of the walls of the upper surface soil into the open borehole during subsequent sampling (Troiano, 1987). The top foot of soil was sampled in 6-inch increments and the remainder in 1-foot increments by combining the contents of two 6-inch cylinders. Basin soil was sampled to a depth of 10 feet, unless prevented by excessive soil moisture. Highway roadside soil was sampled to a depth of 5 feet using a gasoline-powered auger to penetrate the gravel texture of the top 3 feet. The

cylinder weight and the total weight of the cylinder and soil were recorded for bulk density estimation. After mixing in a plastic bag, a 1-pint Mason jar was filled with the sample for chemical analysis and another 150 gram sample was sealed in an 8 by 12-inch plastic bag for texture analysis. Soil texture analyses were conducted on the first core sample collected at each site. Samples submitted for chemical analysis were transported on dry ice and stored frozen until transferred to the laboratory.

SAMPLE EXTRACTION AND ANALYSIS

All chemical analyses of water and soil samples were conducted by the California Department of Food and Agriculture (CDFA), Chemistry Laboratory Services, Environmental Monitoring Section in Sacramento. The complete CDFA Laboratory procedures are contained in Appendix 1.

III. RESULTS AND DISCUSSION

PELTIER BASIN

Inflow canal

Water flowing to the basin in the inflow canal was sampled at four collection sites. Samples from all collection dates were positive. Maximum concentrations were: diuron at 1.42 ppb, oryzalin at 27.40 ppb, oxyfluorfen at 5.62 ppb, and simazine at 83.47 ppb (Table 2).

Stored water

Water stored in the basin was sampled at two collection sites. Maximum concentrations were: diuron at 1.92 ppb, oryzalin at 12.50 ppb, oxyfluorfen at 3.47 ppb, and simazine at 36.18 ppb (Table 3). Water was

Table 2. Concentrations (ppb) of diuron, oryzalin, oxyfluorfen, and simazine in water samples collected from the Peltier basin inflow canal following separate rain events.

Sampling Site #		Dates sampled			
		1/28	2/14	3/5	4/13
		-----ppb-----			
1	diuron	0.65	0.60	0.40	0.44
	oryzalin	- ^a	-	27.40	20.70
	oxyfluorfen	5.62	-	4.61	0.27
	simazine	1.21	3.27	0.29	1.28
2	diuron	1.09	0.53	0.69	1.42
	oryzalin	-	-	8.30	13.40
	oxyfluorfen	2.78	-	0.97	0.26
	simazine	2.66	14.22	9.94	7.80
3	diuron	-	1.00	-	-
	oryzalin	-	-	-	-
	oxyfluorfen	-	-	-	-
	simazine	-	21.56	-	-
4	diuron	0.54	-	0.58	-
	oryzalin	-	-	17.10	-
	oxyfluorfen	4.61	-	3.93	-
	simazine	83.47	-	1.13	-

a) - = No data available.

Table 3. Concentrations (ppb) of diuron, oryzalin, oxyfluorfen, and simazine in samples collected following separate rain events from water stored in the Peltier basin.

		Dates sampled				
		1/28	2/14	3/5	3/26	4/13
		-----inches-----				
		4	24	30	18	18
		-----ppb-----				
Sampling Site #	Water depth					
5	<u>South side</u>					
	diuron	0.80	0.47	1.92	1.49	1.30
	oryzalin	- ^a	-	5.70	2.30	12.50
	oxyfluorfen	3.47	-	0.33	0.10	0.13
	simazine	36.18	14.27	11.19	6.71	3.64
6	<u>North side</u>					
	diuron	-	-	-	1.53	1.13
	oryzalin	-	-	-	1.70	6.30 ^b
	oxyfluorfen	-	-	-	0.11	ND
	simazine	-	-	-	6.68	7.12

a) - = No data available.

b) ND = None detected. Minimum detection limits for diuron, oryzalin, oxyfluorfen, and simazine were .10, .50, .05, and .10 ppb respectively.

stored in the basin continuously from January to mid-April at a depth ranging from 6 to 30 inches. Although most runoff was delivered in the basin inflow canal, runoff was observed to flow directly into the basin from the surrounding ROW, including direct runoff from the Highway 99 roadside treatment strip bordering the basin. Since Caltrans has applied only oryzalin and oxyfluorfen since 1988, roadside application was an unlikely source of simazine or diuron movement.

Water was released from the basin during the March 5 rainstorm, when the water level reached the height of the outflow drain. Basin water samples collected on this date were positive for diuron, oryzalin, oxyfluorfen, and simazine, suggesting the potential for movement of residues to surface waters.

Drywells

Water samples were collected in January and April from the settling chambers of each of the three basin drywells. Maximum concentrations were: diuron at 0.46 ppb, oxyfluorfen at 0.28 ppb, and simazine at 17.88 ppb (Table 4). Drywell concentrations were lower than those detected in basin stored water and in the inflow canal. The drywells were sampled in January as the basin was filling, and in April as the basin was draining. January sampling preceded the end of the soil herbicide treatment season in the agricultural drainage area, and therefore preceded the maximum expected herbicide load in surface water drainage to the basin. Herbicide degradation would be among the factors accounting for low concentrations detected in April.

Table 4. Concentrations (ppb) of diuron, oryzalin, oxyfluorfen, and simazine in water samples collected following separate rain events from the settling chambers of each of the three drywells in the Peltier basin.

		Dates sampled	
		1/29	4/22
Sampling Site #		-----ppb-----	
7	<u>South drywell</u>		
	diuron	0.38	0.42 ^b
	oryzalin	- ^a	ND
	oxyfluorfen	0.28	ND
	simazine	16.69	0.98
8	<u>Middle drywell</u>		
	diuron	0.29	ND
	oryzalin	-	ND
	oxyfluorfen	ND ^c	ND
	simazine	0.68	0.66
9	<u>North drywell</u>		
	diuron	0.39	0.46
	oryzalin	-	ND
	oxyfluorfen	0.14	ND
	simazine	17.88	0.63

a) - = No data available.

b) ND = None detected. Minimum detection limits for diuron, oryzalin, oxyfluorfen, and simazine were .10, .50, .05, and .10 ppb respectively.

c) The MDL for this oxyfluorfen analysis was 2.0 ppb.

Domestic water wells

No herbicides were detected in either of the two domestic wells sampled on properties near the basin's southern perimeter. There were no well logs on record at the California Department of Water Resources (DWR) for these two wells, so construction details are unknown. Mapped water table elevations in 10-foot contour intervals for the area indicated that the direction of ground water flow from the basin was not toward the sampled wells (Figure 7A). Depth to water in wells was estimated at 60-70 feet (Figure 5A).

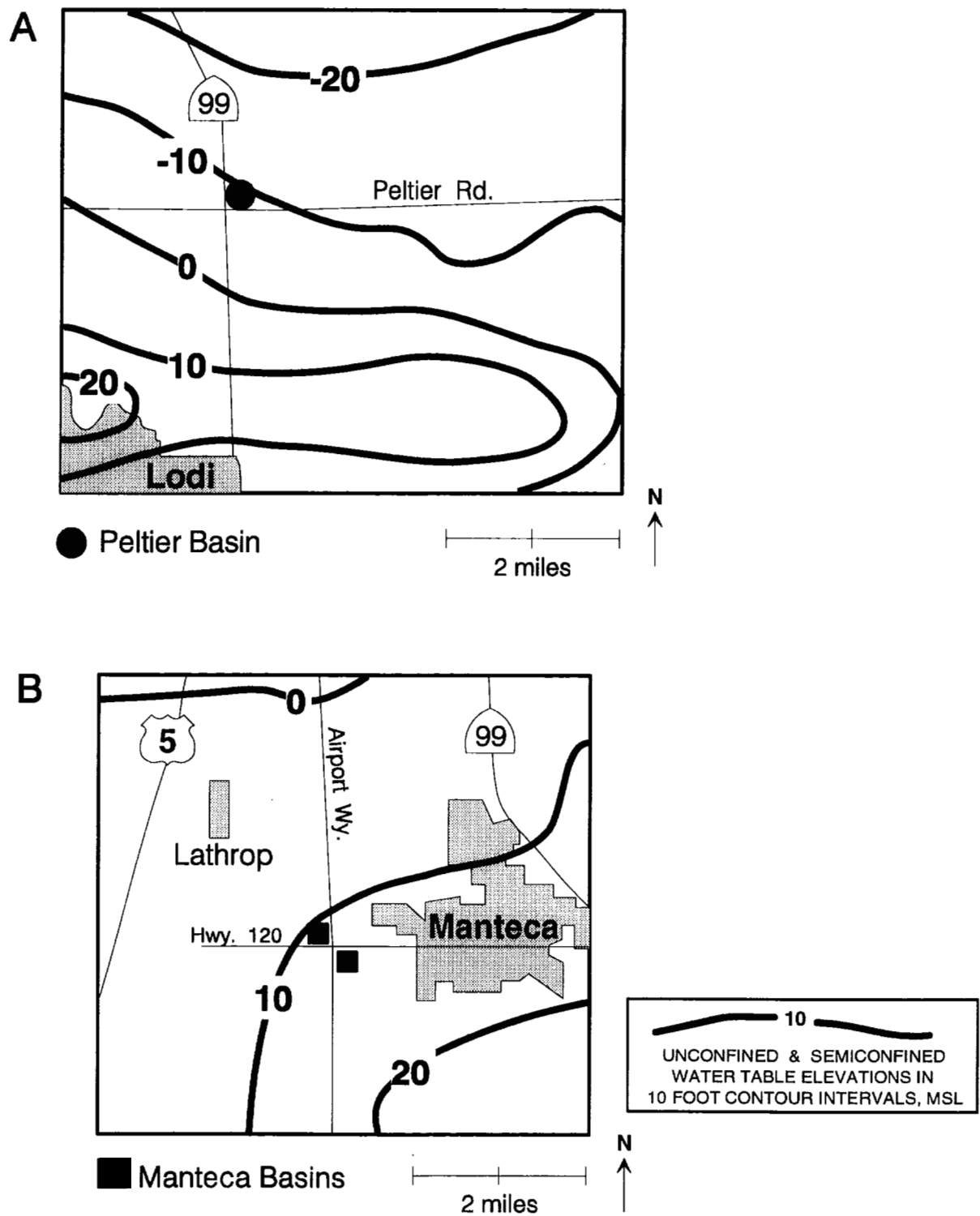
Basin soil

The minimum detectable levels (MDLs) available for chemical analyses of soil mostly were higher than concentrations detected in basin water samples. No residues were detected in either of the two 10-foot soil cores sampled at 1-foot increments. Although information is not available on the current basin infiltration rate, infiltration drainage appeared fairly rapid during final seasonal drainage of the basin in April. Even though basin floor soil-coring was conducted less than two weeks following the final basin drainage, depth of coring was not limited by excessive soil moisture (below 17%) (Appendix 2). Soil texture was coarse and sandy throughout the 10-foot coring depth (Appendix 3).

Highway 99 roadside soil

Soil was sampled from the Highway 99 roadside to detect simazine and diuron residues remaining near the surface from applications prior to 1988. Caltrans had applied only oryzalin and oxyfluorfen to Highway 99 roadsides since 1988. Three 5-foot soil cores were sampled in the

Figure 7. Lines of equal elevation of water in wells for the Peltier and Manteca sites.



Source: San Joaquin County Flood Control and Water Conservation District, Spring 1989

Caltrans treatment strip along Highway 99. Simazine was detected in one of the three cores at depths of 0-12 inches (48.0 ppb), 24-36 inches (24.0 ppb), and at 36-48 inches (14.0 ppb) (Table 5). No diuron was detected. Because of the presence of simazine near the soil surface, past Caltrans treatments to the roadside must be considered a potential contributing source of the simazine residues found in basin water samples. But the absence of diuron in the cores suggests that these residues originated from agricultural drainage.

Oxyfluorfen levels detected in the roadside soil cores were: 25.8 ppb at 0-12 inches in the northern core (260 feet north of Peltier Road); 335.7 ppb at 0-12 inches and 26.9 ppb at 48-60 inches in the middle core (90 feet south of Peltier Road); and 137.9 ppb at 0-12 inches, and 33.7 ppb at 12-24 inches in the southern core (480 feet south of Peltier Road) (Table 5).

MANTECA BASINS

Stored water

Because the Manteca basins receive no surface water runoff from outside the right-of-way, the only potential contributing sources of herbicide movement to the basins were the Caltrans bromacil + diuron treatment to the Highway 120 roadside and the county simazine treatment to the overpass roadside. For the SE basin, only three rainstorms produced sufficient depth of stored water for sampling. All water samples in the SE basin contained bromacil, diuron, and simazine residues. Maximum concentrations detected were: bromacil at 25.62 ppb, diuron at 36.32 ppb, and simazine at 78.80 ppb. For the NW basin, only

Table 5. Concentrations (ppb) of diuron, oxyfluorfen, and simazine in soil core samples collected from the Caltrans roadside treatment strip along Highway 99 in the Peltier basin drainage area.

Sampling

Site #

14 Southern core (5/20/92)

	<u>diuron</u>	<u>oxyfluorfen</u>	<u>simazine</u>
Depth (inches)			
0-12	ND ^a	137.9	ND
12-24	ND	33.7	ND
24-36	ND	ND	ND
36-48	ND	ND	ND
48-60	ND	ND	ND

15 Middle core (5/20/92)

	<u>diuron</u>	<u>oxyfluorfen</u>	<u>simazine</u>
Depth (inches)			
0-12	ND	335.7	48.0
12-24	ND	ND	ND
24-36	ND	ND	24.0
36-48	ND	ND	14.0
48-60	ND	26.9	ND

16 Northern core (5/20/92)

	<u>diuron</u>	<u>oxyfluorfen</u>	<u>simazine</u>
Depth (inches)			
0-12	ND	25.8	ND
12-24	ND	ND	ND
24-36	ND	ND	ND
36-48	ND	ND	ND
48-60	ND	ND	ND

a) ND = None detected. Minimum detection limits for diuron, oxyfluorfen, and simazine were 40.0, 40.0, 20.0, and 4.0 ppb respectively.

two rainstorms produced sufficient depth of stored water for sampling. Maximum concentrations detected were: bromacil at 2.55 ppb, diuron at 0.85 ppb, and simazine at 1.10 ppb (Table 6).

The SE basin appeared to collect a greater volume of runoff than the NW basin following each rainstorm. A graded dirt ramp in the SE basin, extending from the roadside to the basin floor, appeared to channel runoff into the basin.

Domestic water wells

No herbicides were detected in the domestic well sampled on private property near the southern perimeter of the SE basin. There was no well log on record at DWR, so construction details are unknown. Depth to water in wells in the area of the basins was estimated at 10-20 feet (Figure 5B). The water table was reached during soil coring at a soil depth of 30 inches in the NW basin (January and April) and at 48 inches in the SE basin (April). Although the water table is shallow, DWR well logs for other wells in the same section indicate domestic water well depths greater than 100 feet.

Basin soil

The single background core taken from the NW basin was limited to a depth of 60 inches due to the shallow water table. No residues were detected. Soil core samples were taken from both basins following final seasonal drainage of stored water in April. The shallow water table limited soil coring depths to 30 inches in the Manteca NW basin and to 48 inches in the SE basin. Although bromacil, diuron, and simazine were detected in

Table 6. Concentrations (ppb) of bromacil, diuron, and simazine in samples collected following separate rain events from water stored in the Manteca basins.

Manteca NW basin						
		Dates sampled				
		1/29	2/14	3/6	3/26	4/13
		-----inches-----				
Water depth		<1	<1	4	<1	<1
Sampling Site #		-----ppb-----				
17						
bromacil		- ^a	-	2.55	-	ND ^b
diuron		-	-	0.85	-	0.77
simazine		-	-	0.53	-	1.10

Manteca SE basin						
		Dates sampled				
		1/29	2/14	3/6	3/26	4/13
		-----inches-----				
Water depth		<1	4	6	<1	4
Sampling Site #		-----ppb-----				
19						
bromacil		-	25.62	3.74	-	0.52
diuron		-	36.32	12.10	-	1.61
simazine		-	78.80	16.85	-	11.22

a) - = No data available.

b) ND = None detected. Minimum detection limits for bromacil, diuron, and simazine were .25, .10, and .10 ppb respectively.

water samples from each basin, only simazine was detected in soil. Simazine was found in the SE basin core at depths of 0-6 inches (36.0 ppb) and 12-24 inches (15.0 ppb)(Table 7). No herbicides were detected in the NW basin core. Because herbicide leaching is to some extent a function of water solubility, the relatively more water-soluble bromacil and diuron would be expected to leach to a greater extent than the less water-soluble simazine, which may explain the presence of simazine and the absence of bromacil and diuron residues. Water solubility values (ppm) are 820, 42, and 4.9 for bromacil, diuron, and simazine respectively (Johnson, 1991).

A single water sample was collected (April 27) from seepage into the bottom of the soil core borehole in the SE basin (48-inch depth); simazine was detected at 1.0 ppb (Table 8). Water and soil sample data suggest that County-applied simazine was transported in runoff to the Manteca basins where it percolated to underlying soil.

Table 7. Concentrations (ppb) of bromacil, diuron, and simazine in soil core samples collected from the Manteca basins following final seasonal drainage of stored water.

Manteca NW basin (4/27/92)

Sampling
Site #
18

	<u>bromacil</u>	<u>diuron</u>	<u>simazine</u>
Depth (inches)			
0-6	ND ^a	ND	ND
6-12	ND	ND	ND
12-24	ND	ND	ND
24-36	ND	ND	ND
36-48	ND	ND	ND

Manteca SE basin (4/27/92)

Sampling
Site #
20

	<u>bromacil</u>	<u>diuron</u>	<u>simazine</u>
Depth (inches)			
0-6	ND	ND	36.0
6-12	ND	ND	ND
12-24	ND	ND	15.0
24-36	ND	ND	ND
36-48	ND	ND	ND

a) ND = None detected. Minimum detection limits for bromacil, diuron, and simazine were 40.0, 40.0, and 4.0 ppb respectively.

IV. CONCLUSIONS

Results of water and soil monitoring are summarized in Tables 8 and 9. Detection of herbicide residues in water sampled from storm runoff flowing into a basin, and in water sampled from basins and basin drywells, indicates a potential for the herbicides to be transported to ground water by soil infiltration of water accumulated in the basins. According to the herbicide active ingredients detected at the Peltier basin, the source of residues was from runoff from Caltrans roadside applications and from agricultural properties. Simazine and diuron residues were probably transported from properties outside the right-of-way, because Caltrans had applied only oryzalin and oxyfluorfen to the roadside since 1988. The oryzalin and oxyfluorfen residues may have been transported either from rights-of-way applications or from adjacent properties. Detection of herbicides other than those used exclusively for rights-of-way weed control suggests further investigation of the contribution of herbicide transport in runoff from properties adjacent to rights-of-way basins. Herbicide residues detected at the Manteca basins were solely attributed to rights-of-way treatments from Caltrans applications of bromacil and diuron, and from County applications of simazine. Rapid movement from surface to subsurface layers in infiltration basins represents a potential pathway for ground water contamination.

Table 8. Summary of herbicide concentrations (ppb) found in water samples collected at the Peltier and Manteca sites.

PELTIER		
	mean (ppb)	range (ppb)
Inflow canal		
diuron	0.72	0.40 to 1.42
oryzalin	17.38	8.30 to 27.40
oxyfluorfen	2.88	0.26 to 5.62
simazine	13.35	0.29 to 83.47
Stored water		
diuron	1.23	0.47 to 1.92
oryzalin	5.70	1.70 to 12.50
oxyfluorfen	0.83	0.10 to 3.47
simazine	12.26	3.64 to 36.18
Drywells		
diuron	0.39	0.29 to 0.46
oryzalin	ND ^a	
oxyfluorfen	0.21	0.14 to 0.28
simazine	6.27	0.63 to 17.88
Domestic water wells	ND	
MANTECA		
Stored water		
NW basin		
bromacil	2.55	(one sample)
diuron	0.81	0.77 to 0.85
simazine	0.82	0.53 to 1.10
SE basin		
bromacil	9.96	0.52 to 25.62
diuron	16.68	1.61 to 36.32
simazine	35.62	11.22 to 78.80
Domestic water well	ND	
Soil core borehole water sample		
SE basin		
bromacil	ND	
diuron	ND	
simazine	1.00	(one sample)

a) ND = None detected. Minimum detection limits for bromacil, diuron, oryzalin, oxyfluorfen, and simazine were .25, .10, .50, .05, and .10 respectively, and 0.20 for bromacil, diuron, and simazine in the soil core borehole water sample.

Table 9. Summary of herbicide concentrations (ppb) found in soil samples collected at the Peltier and Manteca sites.

PELTIER			
		mean (ppb)	range (ppb)
Basin floor	(April)	ND ^a	
Highway 99 roadside	(May)		
diuron		ND	
oxyfluorfen		112.0	25.8 to 335.7
simazine		28.67	14.0 to 48.0
MANTECA			
		mean (ppb)	range (ppb)
Basin floor			
NW basin	(January)	ND	
NW basin	(April)	ND	
SE basin	(April)		
bromacil		ND	
diuron		ND	
simazine		25.5	15.0 to 36.0

a) ND = None detected. Minimum detection limits for bromacil, diuron, oxyfluorfen, and simazine were 40.0, 40.0, 20.0, and 4.0 ppb respectively.

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APPENDICES

APPENDIX 1.

SAMPLE EXTRACTION AND ANALYSIS METHODS

CALIFORNIA DEPT. OF FOOD & AGRIC.
CHEMISTRY LABORATORY SERVICES
ENVIRONMENTAL MONITORING SECTION
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Original Date: 06/09/91
Supercedes: New
Current Date: 12/08/92
Method #:

OXYFLUORFEN IN SURFACE WATER

SCOPE:

This method is for the determination of Oxyfluorfen (Goal) in surface water.

PRINCIPLE:

The samples of water were extracted by shaking in a separatory funnel with methylene chloride. The extract was filtered and evaporated to dryness. It was then transferred and brought up to final volume with hexane. The extract was analyzed by gas chromatography using a flame photometric detector (FPD).

REAGENTS AND EQUIPMENT:

Methylene chloride and hexane (pesticide residue grade)
Sodium sulfate (anhydrous)
Separatory funnels (2 L)
Boiling flasks, flat bottom with ground glass joint 24/40 (500 mL)
Glass stem funnels (65 mm/75 mm)
Rotary evaporator (Büchi/Brinkmann, R110)
Graduate test tubes (15 mL)
Nitrogen evaporator (Organomation Model # 12)
Vortex mixer for test tubes
Balance (Mettler PC 4400)
Filter paper (Whatman #1, 12.5 cm)

ANALYSIS:

- 1) Remove samples from refrigerated storage and allow them to come to room temperature. Samples consist of approximately 1 L and are stored in 1 L amber glass bottles to prevent any photodegradation from occurring.
- 2) Record weight of the sample by weighing sample bottle before and after transfer.
- 3) Extract sample by shaking with 100 mL of methylene chloride for 2 min.
- 4) Allow layers to separate and filter the organic layer through 25 g anhydrous sodium sulfate and filter paper. Collect extract in a 500 mL boiling flask.
- 5) Repeat steps 3 & 4 two more times using 80 mL of methylene chloride each time.

- 6) Rinse sodium sulfate with 20 mL additional methylene chloride and collect in the same 500 mL boiling flask.
- 7) Take extract almost to dryness on a rotary evaporator. Add a 3-4 mL hexane to the flask. Concentrate to 1-2 mL.
- 8) Transfer extract to a graduated test tube. Rinse flask 3 times each with 2 mL of hexane. Transfer each wash to the same graduated test tube.
- 9) Place extract in a nitrogen evaporator with waterbath set at 40°C and evaporate to a final volume of 1 mL under a gentle stream of nitrogen.
- 10) Stopper the graduated test tube and mix contents by placing on a vibrating mixer for about 15 seconds. Submit sample for gas chromatographic analysis.

EQUIPMENT CONDITIONS:

PRIMARY ANALYSIS

HP-5890 GC with ECD

Column: ULTRA 1 (100% dimethyl polysiloxane) 25 m x 0.2 mm x 0.33 um

Carrier gas: Helium, Flow rate: 20 mL/min.

Injector: 200°C.

Detector: 250°C.

Temperature program: Initial Temperature: 175°C

Initial Time: 1 min

Rate: 25°C/min

Final Temperature: 250°C

Final Time: 5 minutes

Injection volume: 2 uL

Retention times: 7.50 minutes

Linearity checked: 0.2 ng - 20 ng

CONFIRMATION ANALYSIS

Varian: 3700 GC WITH HALL Detector

Column: HP-17 (50% Phenyl and 50% methyl polysiloxane) 10 m x 0.53 mm
x 0.2 um

Carrier gas: Helium, Flow rate: 17 mL/min.

Injector: 220°C.

Detector: 260°C.

Temperature program: Initial Temp: 150°C held for 2 minutes.

Rate: 25°C/minute.

Final Temp: 250°C held for 3 minutes.

Injection volume: 2 uL

Retention times: 7.750 min

Linearity checked: 0.2 ng - 20 ng

CONFIRMATION ANALYSIS

Hewlett Packard 5890 A GC with FPD

Column: DB-1701 (7% cyanopropyl 7% polysiloxane) 30 m x 0.25 mm x 0.25 um

Carrier gas: Helium; Flow rate: 20 mL/min.

Injector: 220°C.

Detector: 250°.

Temperature program: Initial Temp: 150°C held for 2 minutes.
 Rate: 25°C/minute.
 Final Temp: 250°C held for 3 minutes.
 Injection volume: 2 uL
 Retention times: 10.370 min
 Linearity checked: 0.2 ng - 20 ng

CALCULATIONS:

PPB OXYFLUORFEN

$$\text{ppb in sample} = \frac{(\text{peak height sample})(\text{ng/uL std})(\text{uL injected std})(\text{final volume mLs})(1000)}{(\text{peak height std})(\text{uL injected sample})(\text{weight of sample g})}$$

FORTIFICATION:

Oxyfluorfen was spiked into separate 1 L volumes of water
 at the levels listed below.

RECOVERIES:

% Recoveries of oxyfluorfen

Levels	Oxyfluorfen (mean)	% CV
0.1 ppb (n=5)	97.8	6.6
0.25 ppb (n=5)	106.6	7.3
0.50 ppb (n=5)	100.1	10.1

Recovery validation was done prior to samples.

MINIMUM DETECTABLE LEVEL:

The minimum detectable level was 0.05 ppb (1 liter volume of sample used.)
 S/N=4

DISCUSSION:

At the beginning and end of each run standards were run consisting of 0.1, 0.25, 0.5, and 1.0 ng/uL.

REFERENCE:

- 1) White, Jane, *Diazinon, Chlorpyrifos, Parathion and Methidathion In Fog Water*, 1989, Environmental Monitoring Methods, California Department of Food and Agriculture.

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Original Date: March 4, 1992
Supersedes:
Current Date: March 4, 1992
Method#:

HPLC Determination of Surflan in Water

SCOPE:

This method determines surflan residue in water with a minimum detection limit of 0.5 ppb.

PRINCIPLE:

The water samples are extracted with methylene chloride. After evaporating the solvent, the extracts are dissolved in methanol and analyzed by HPLC on a C-18 reversed phase column. The UV detector is set at 280 nm.

REAGENT AND EQUIPMENT:

Methanol, residue grade.
Methylene Chloride, residue grade.
Anhydrous sodium sulfate, residue grade.
Boiling flasks, flat bottomed (500mL).
Separatory funnel, 1 L.
Rotary evaporator (Büchi/Brinkmann, R110).
Nitrogen sweep evaporator (Organomation Model #12)
Disposable filters, 0.2 micron (Nylon Acrodisc)
HPLC, Perkin-Elmer series 4
UV detector, Varian 2550.

EXTRACTION:

1. Allow sample to equilibrate to ambient temperature. Measure 800 mL (or by weight) of the sample to be analyzed into a one-liter separatory funnel and record the volume or the weight to one decimal point.
2. Add 100 mL of methylene chloride and stopper the funnel. Shake vigorously for one minute.
3. Let the funnel set for approximately 5 minutes or longer allowing the two phases to separate.
4. Drain the lower phase into a 500 mL flat bottom boiling flask through a 9 cm funnel containing 10-15 grams of anhydrous sodium sulfate supported by a bed of glass wool.

5. Repeat steps 2-4 twice with 80 mL of methylene chloride each time. Wash the sodium sulfate bed with an additional 20 mL methylene chloride. Combine the extracts and the wash.

6. Evaporate the solvent on a rotary vacuum evaporator at 30-35°C with 20 inches vacuum.

7. Add 5-8 mL methanol to the flask and swirl gently to allow any residue to dissolve. Filter the solution through a 0.2 micron Acrodisc using a hypodermic syringe and collect the filtrate into a precalibrated 15 mL conical centrifuge tube. Rinse the flask twice with 1-2 mL methanol each time and filter through the same unit.

8. Evaporate the the solution to 5 mL in a 30°C water bath with a gentle stream of nitrogen. Transfer 1.5 mL of the solution to an auto sampler vial for analysis.

INSTRUMENT CONDITIONS:

Perkin-Elmer series 4 HPLC with autosampler and Varian 2550 UV detector.

Column: Beckman Ultrasphere C-18 reversed phase, 15 cm x 4.6 mm , 5 μ .

Sample injection volume: 20 μ L.

Flow rate: 1 mL/min.

Mobile phase: 50% methanol and 50% water.

Detector: 280 nm.

RECOVERY:

<u>SPIKE LEVEL</u> (ppb)	<u>FINDING</u> (ppb)	<u>RECOVERY</u> (%)	<u>STANDARD</u> <u>DEVIATION</u>	<u>n</u>
1.0	0.886	88.6	0.147	5
2.5	2.16	86.4	0.113	5
5.0	4.29	85.8	0.583	5

REFERENCE:

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Kennedy, J. H., "High Pressure Liquid Chromatographic Analysis of Oryzalin," J. of Chromatographic Science, Volume 15, pp 79-81, 1977

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TITTLE: SUPERVISING CHEMIST

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Original Date:03/24/1990
Supersedes: NEW
Current Date:04/10/1990
Method #:

**MULTIPESTICIDE RESIDUE ANALYSIS:
ATRAZINE, BROMACIL, DIURON, PROMETON, SIMAZINE IN WELL WATER.**

SCOPE:

This method is developed to analyze Atrazine, Bromacil, Diuron, Prometon, and Simazine in well water.

PRINCIPLE:

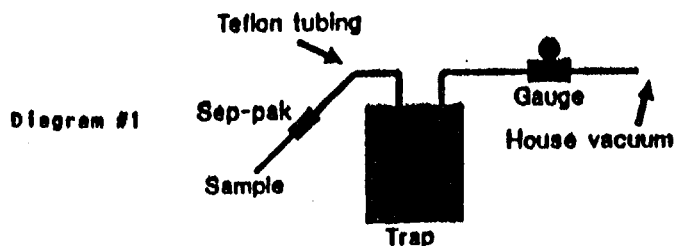
A conditioned C 18 reversed phase Sep-pak is used to trap Atrazine, Bromacil, Diuron, Prometon and Simazine from water samples. The Sep-pack is then centrifuged to eliminate any remaining water. Methanol is then used to elute all chemicals. The eluant is then concentrated and analyzed for Diuron and Bromacil by LC, for Atrazine, Prometon, Simazine by GC.

REAGENTS AND EQUIPMENT:

Methanol, pesticide grade or equivalent.
Distilled water.
Working standards in Methanol (Diluted from stock standard.)
In house vacuum manifold.
In house aspiration system.
C18 reversed phase Sep-pak, [®]Water Division of Millipore.
Nylon acrodisc, 0.2 micron, Gelman Sciences.
Centrifuge: Clay Adams.
Beakers, 600 mL.
Graduated test tubes, 10 mL.
Micro-Mate Syringes, 10 cc - Popper & Sons Inc.
N-EVAP - Meyers Organomation Associates Incorporated
Vibrating mixer.
Sodium Sulfate, anhydrous, granular (ACS).

ANALYSIS:

1. For each sample, weigh 500.0 grams of water sample into two separated 600ml beakers.
2. Connect a C 18 reversed phase Sep-pak to the in house vacuum manifold as follows in diagram #1.



ANALYSIS:

3. Condition the Sep-pak with about 5 mL of methanol followed by about 10 mL of distilled water by applying in house vacuum. Do not let the sep-pak go to dryness.
 4. Attach the conditioned Sep pak to a 15 mm glass tubing and dip into the beaker containing the 500g of sample. Adjust the flow rate to about 3-5 ml/minute (about 6 in Hg).
 5. After all 500g of water sample has passed through the Sep-pak, leave the vacuum on for few minutes.
 6. Remove the Sep-pak and insert the sep-pak into a centrifuge tube and centrifuge for 1 minute at 1200 rpm by setting the dial at 4 on the centrifuge.
 7. Elute all chemicals with 8 mL of methanol by using the in house aspiration system into a 10 mL graduate test tube.
 8. Concentrate the eluting solvent to 1.0 mL by using the Nitrogen evaporator. Mix it well for 30 seconds by using the vibrating mixer. Filter through a 0.2 um acrodisc into three separated micro vials.
- Analyze by gas chromatograph and liquid chromatograph*

EQUIPMENT CONDITIONS:

- A. Gas chromatograph: HP 3700 with TSD.
Column: HP-17 10 m x 0.53 mm. Film thickness: 2.0 um.
Temperature program: Isothermal 175°C.
 Injector: 220°C, detector: 220°C.
Carrier gas: Helium. Flow rate: 20 mL/min.
Sample injected: 2 ul.
Retention times: Prometon ~ 2.40 minutes
 Atrazine ~ 2.82 minutes
 Simazine ~ 3.04 minutes
- B. Liquid chromatography: Perkin Elmer Series 4.
Column: BECKMAN ODS, 5.0 um, 4.6 mm x 15.0 cm.
Guard column: BECKMAN ODS, 5.0 um, 4.6 mm x 4.5 cm.
Detector: Varian 2550 UV.
Flow rate: 1.0 ml/min.
Sample injected: 60 ul.

For Diuron analysis:

Mobile phase: 55% water, 45% acetonitrile.

Wave length: 254 nm.

Retention time: ~ 5.60 minutes.

For Bromacil analysis:

Movile phase: 70% water, 30% acetonitrile.

Wave length: 280 nm.

Retention time: ~ 5.14 minutes.

CONFIRMATION: Atrazine, Prometon and Simazine are confirmed by Varian 6000 with TSD. Column: 20 m x 0.53 mm x 1.3 um Carbowax. Injector: 220°C, detector: 220°C. Temperature program: Int: 150°C.

Int time: 0 min.

Rate: 15°C/min.

Final time: 9 min.

Carrier gas: Helium. Flow rate: 25 mL/min.

Retention times: Prometon ~ 5.7 minutes.

Atrazine ~ 7.8 minutes.

Simazine ~ 9.3 minutes.

Bromacil is confirmed by TSD/DB-1301 30 m x 0.53 mm x 1.0 um column.

Carrier gas: Helium. Flow rate: 25 mL/min.

Isothermal 190°C, injector: 220°C, detector: 220°C.

Retention time: ~ 8.9 minutes.

Diuron is not confirmed at MDL level.

CALCULATIONS:

$$\text{PPB} = \frac{\text{Peak height of sample} \times \text{Amount of std (ng)} \times 1,000 \mu\text{l}}{\text{Peak height of std} \times \text{volume injected} \times \text{sample weight (g)}}$$

DISCUSSION:

Minimum detection limit (Signal to noise ratio is 5 to 1.) for these chemicals by this method was 0.1 ppb.

DIODE ARRAY DETECTOR was tried to analyze bromacil and diuron. However, the sensitivity did not meet the requirement.

The diagram #1 is a in house system. If you have any question about it, please contact the above address.

The following results were obtained from different spike levels by multipoints calibration method:

Chemical	Spike level (ppb)	Number of analysis (n)	Mean % Recovery	Standard deviation (+/-)
Atrazine	4.0	5	102.7	7.9
Prometon	4.0	5	105.5	9.6
Simazine	4.0	5	107.4	8.8
Bromacil	4.0	5	103.5	6.2
Diuron	4.0	5	102.2	4.7

DISCUSSION:

Chemical	Spike level (ppb)	Number of analysis (n)	Mean % Recovery	Standard Deviation (+/-)
Atrazine	2.0	5	90.4	3.5
Prometon	2.0	5	91.5	4.8
Simazine	2.0	5	89.4	6.6
Bromacil	2.0	5	87.7	6.8
Diuron	2.0	5	88.2	7.2
Atrazine	0.5	10	106.8	13.3
Prometon	0.5	10	103.0	6.9
Simazine	0.5	10	105.6	15.6
Bromacil	0.5	10	92.0	9.7
Diuron	0.5	10	99.6	14.8

REFERENCES:

WRITTEN BY: Duc Tran

Duc Tran
TITLE: Agricultural Chemist I

REVIEWED BY: Catherine Cooper

Catherine Cooper
TITLE: Agricultural Chemist III

APPROVED BY: S. Mark Lee

S. Mark Lee
TITLE: Principal Investigator

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ENVIRONMENTAL MONITORING SECTION
3292 Meadowview Road
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Original Date:
Supercedes: New
Current Date: 12/09/92
Method #:

ANALYSIS OF OXYFLUORFEN IN SOIL

SCOPE:

This method is for the determination of Oxyfluorfen (Goal) in soil.

PRINCIPLE:

The chemicals are extracted from soil with a mixture of hexane:acetone. An aliquot is concentrated to eliminate acetone (Azeotrope: 49.8°C) and then transferred to a pre-conditioned silica gel Sep-pak[®]. After the Sep-pak[®] is washed with hexane, all chemicals are eluted with methanol.

REAGENTS AND EQUIPMENT:

Acetone, pesticide grade
Hexane, pesticide grade
Methanol, pesticide grade
Sodium Sulfate, anhydrous, granular (ACS)
Bottles, 500 mL amber wide-mouth with lid
Graduate cylinder, 100 mL
Funnels, 60° short stem, 3-4 inch diameter
Graduate test tube, 15 mL
Whatman #1 filter paper, 12.5 cm
Micro-Mate[®] Syringes, 10 cc - Popper & Sons Inc.
Nylon Acrodisc[®], 0.2 micron, Gelman Sciences
Sep-pak[®] silica gel, Waters and Associates
Balance - Mettler PL 1200, Mettler Instrument Corp.
G-10 Gyrotory[®] Shaker with CE-250S clamps, New Brunswick Scientific Co., Inc.
Evaporator with nitrogen blow-down, (Model #12), Organomation Associates Inc.
Vortex mixer
Centrifuge, Clay Adams (Model #0005)
Pipette
Vac Elut SPS 24 (vacuum manifold), Analytichem International

ANALYSIS:

- 1) Weigh 25 g of soil into a 500 mL brown bottle. Add 30 g of sodium sulfate and 50 mL of a hexane:acetone (60:40) mixture.
- 2) The sample bottle was shaken for two hours at 210 rpm on a mechanical shaker.
- 3) Decant the extract through a funnel containing filter paper and 20 g sodium sulfate into a 100 mL graduated cylinder.

- 4) Add 20 mL of the hexane:acetone (60:40) mixture to the brown bottle and shake it for 1-2 minutes. Decant the extract into the cylinder. Wash the funnel with about 10 mL of hexane:acetone (40:60) and bring the volume to 75 mL with the mixture.
- 5) Pipet 15 mL of the extract into a graduated test tube. Concentrate to 1.0 mL using a nitrogen evaporator set at 45°C. Add 1 mL of hexane to the test tube and 2 g of anhydrous sodium sulfate. Mix well on a vortex mixer.
- 6) Connect a silica gel Sep-pak[®] to a 10 mL syringe. Attach the syringe and Sep-pak to a Vac Elute SPS 24. Condition the Sep-pak[®] by adding 4 mL of hexane and slowly pressing the plunger to obtain a flow rate about 3 mL/min. Maintain this flowrate if possible.
- 7) Quantitatively transfer the extract from the test tube to the syringe with the conditioned silica gel Sep-pak[®]. Pass the extract through the Sep-pak[®] discarding the solvent.
- 8) Wash the Sep-pak[®] with 4 mL of hexane. Discard the expelled solvent. Centrifuge the Sep-pak[®] at 1100 rpm about for 30 seconds.
- 9) Reconnect the Sep-pak[®] to the syringe and add 10 mL of methanol to the syringe and elute. Collect the sample extract in a graduated test tube.
- 10) Concentrate the eluant to 1 mL, using the evaporator with nitrogen. Filter through an Acrodisc[®] into 2 autosampler vials. Analyze Prometone, Atrazine and Simazine by GC/NPD. Bromacil and Diuron are analyzed by HPLC/UV.

EQUIPMENT CONDITIONS:

PRIMARY ANALYSIS

HP-5890 GC with ECD

Column: ULTRA 1 (100% dimethyl polysiloxane) 25 m x 0.2 mm x 0.33 um

Carrier gas: Helium, Flow rate: 20 mL/min.

Injector: 200°C.

Detector: 250°C.

Temperature program: Initial Temperature: 175°C

Initial Time: 1 min

Rate: 25°C/min

Final Temperature: 250°C

Final Time: 5 minutes

Injection volume: 2 uL

Retention times: 7.50 minutes

Linearity checked: 0.2 ng - 20 ng

CONFIRMATION ANALYSIS

Varian: 3700 GC WITH HALL Detector

Column: HP-17 (50% Phenyl and 50% methyl polysiloxane) 10 m x 0.53 mm
x 0.2 um

Carrier gas: Helium, Flow rate: 17 mL/min.

Injector: 220°C.

Detector: 260°C.

Temperature program: Initial Temp: 150°C held for 2 minutes.

Rate: 25°C/minute.

Final Temp: 250°C held for 3 minutes.

Injection volume: 2 uL

Retention times: 7.750 min

Linearity checked: 0.2 ng - 20 ng

CONFIRMATION ANALYSIS

Hewlett Packard 5890 A GC with FPD

Column: DB-1701 (7% cyanopropyl 7% polysiloxane) 30 m x 0.25 mm x 0.25 µm

Carrier gas: Helium; Flow rate: 20 mL/min.

Injector: 220°C.

Detector: 250°.

Temperature program: Initial Temp: 150°C held for 2 minutes.

Rate: 25°C/minute.

Final Temp: 250°C held for 3 minutes.

Injection volume: 2 uL

Retention times: 10.370 min

Linearity checked: 0.2 ng - 20 ng

CALCULATIONS:

PPB OXYFLUORFEN

$$\text{ppb in sample} = \frac{(\text{peak height sample})(\text{ng/uL std})(\text{uL injected std})(\text{final volume mLs})(1000)}{(\text{peak height std})(\text{uL injected sample})(\text{weight of sample g})}$$

FORTIFICATION:

Oxyfluorfen was spiked into separate 1 L volumes of water at the levels listed below.

RECOVERIES:

% Recoveries of oxyfluorfen

Levels	Oxyfluorfen (mean)	% CV
40 ppb (n=5)	92.1	4.0
100 ppb (n=5)	94.0	7.0
200 ppb (n=5)	85.2	2.5

Recovery validation was done prior to samples.

MINIMUM DETECTABLE LEVEL:

The minimum detectable level was 40.0 ppb (25 g sample used) S/N=4.

DISCUSSION:

At the beginning and end of each run standards were run consisting of 0.1, 0.25, 0.5, and 1.0 ng/uL.

REFERENCE:

Duc Tran, Residue Analysis Of Atrazine in soil. 1989.

WRITTEN BY: Jorge L. Hernandez

Jorge L. Hernandez

TITLE: Agricultural Chemist I

REVIEWED BY: Catherine Cooper

Catherine Cooper

TITLE Agricultural Chemist III

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Original Date: 07/20/90
Supercedes:
Current Date: 09/04/90
Method #:

RESIDUE ANALYSIS OF TRIAZINES IN SOIL

SCOPE:

This method was developed for the chemical analysis of Bromacil, Diuron, Prometone, Atrazine, and Simazine in soil.

PRINCIPLE:

The chemicals are extracted from soil with a mixture of hexane:acetone. An aliquot is concentrated to eliminate acetone (Azeotrope: 49.8°C) and then transferred to a pre-conditioned silica gel Sep-pak[®]. After the Sep-pak[®] is washed with hexane, all chemicals are eluted with methanol.

REAGENTS AND EQUIPMENT:

Acetone, pesticide grade
Hexane, pesticide grade
Methanol, pesticide grade
Sodium Sulfate, anhydrous, granular (ACS)
Bottles, 500 mL amber wide-mouth with lid
Graduate cylinder, 100 mL
Funnels, 60° short stem, 3-4 inch diameter
Graduate test tube, 15 mL
Whatman #1 filter paper, 12.5 cm
Micro-Mate[®] Syringes, 10 cc - Popper & Sons Inc.
Nylon Acrodisc[®], 0.2 micron, Gelman Sciences
Sep-pak[®] silica gel, Waters and Associates
Balance - Mettler PL 1200, Mettler Instrument Corp.
G-10 Gyrotory[®] Shaker with CE-250S clamps, New Brunswick Scientific Co., Inc.
Evaporator with nitrogen blow-down, (Model #12), Organomation Associates Inc.
Vortex mixer
Centrifuge, Clay Adams (Model #0005)
Pipette

ANALYSIS:

- 1) Weigh 25 g of soil into a 500 mL brown bottle. Add 30 g of sodium sulfate and 50 mL of a hexane:acetone (60:40) mixture.
- 2) The sample bottle was shaken for two hours at 210 rpm on a mechanical shaker.
- 3) Decant the extract through a funnel containing filter paper and 20 g sodium sulfate into a 100 mL graduated cylinder.

- 4) Add 20 mL of the hexane:acetone (60:40) mixture to the brown bottle and shake it for 1-2 minutes. Decant the extract into the cylinder. Wash the funnel with about 10 mL of hexane:acetone (40:60) and bring the volume to 75 mL with the mixture.
- 5) Pipet 15 mL of the extract into a graduated test tube. Concentrate to 1.0 mL using a nitrogen evaporator set at 45°C. Add 1 mL of hexane to the test tube and 2 g of anhydrous sodium sulfate. Mix well on a vortex mixer.
- 6) Connect a silica gel Sep-pak[®] to a 10 mL syringe. Condition the Sep-pak[®] by adding 4 mL of hexane and slowly pressing the plunger to obtain a flow rate about 3 mL/min. Maintain this flowrate if possible
- 7) Quantitatively transfer the extract from the test tube to the syringe with the conditioned silica gel Sep-pak[®]. Pass the extract through the Sep-pak[®] discarding the solvent.
- 8) Wash the Sep-pak[®] with 4 mL of hexane. Discard the expelled solvent. Centrifuge the Sep-pak[®] at 1100 rpm about for 30 seconds.
- 9) Reconnect the Sep-pak[®] to the syringe and add 10 mL of methanol to the syringe and elute. Collect the sample extract in a graduated test tube.
- 10) Concentrate the eluant to 3 mL, using the evaporator with nitrogen. Filter through an Acrodisc[®] into 2 autosampler vials. Analyze Prometone, Atrazine and Simazine by GC/NPD. Bromacil and Diuron are analyzed by HPLC/UV.

EQUIPMENT CONDITIONS:

PRIMARY ANALYSIS:

Gas chromatograph: Varian 6000 with TSD

Column: HP-Carbowax 20M (polyethylene glycol) 30 m x 0.53 mm x 1.33 um

Carrier gas: Helium: Flow rate: 20 mL/min

Injector: 210°C

Detector: 250°C

Temperature program: Initial temp: 130°C

Rate: 15°C/min

Level 1 temp: 190°C

Hold time: 0 min

Final temp: 220°C

Rate: 25°C/min

Hold time: 1 min

Sample injected: 2 uL

Retention times: Prometone - 3.2 min

Atrazine - 4.1 min

Simazine - 4.5 min

Linearity checked: 0.2 ng - 20 ng

Liquid chromatograph: Perkin Elmer Series 4

Column: Beckman ODS, 5.0 μ m, 4.6 mm x 25.0 cm

Guard column: Beckman ODS, 5.0 μ m, 4.6 mm x 4.5 cm

Detector: Varian 2550 UV

Flow rate: 1 ml/min

Sample injected: 40 μ l

For Diuron analysis:

Mobile phase: 55% Water, 45% Acetonitrile

Wavelength: 254 nm

Retention time: Diuron = 5.6 min

Linearity checked: 0.2 ng - 100 ng

For Bromacil analysis:

Mobile phase: 70% Water, 30% Acetonitrile

Wavelength: 280 nm

Retention time: Bromacil = 5.14 min

Linearity checked: 0.2 ng - 100 ng

CONFIRMATION ANALYSIS:

Gas Chromatograph: Varian 3700 GC with FPD

Column: HP-17 (50% phenyl, 50% methyl-polysiloxane) 10 m x 0.53 mm
x 2.0 μ m

Carrier gas: Helium, flow rate: 15 mL/min

Injector: 200°C

Detector: 250°C

Temperature program: Initial temp: 175°C held for 5 min

Rate: 35°C/min

Final temp: 220°C held for 4 min

Injection volume: 2 μ L

Retention times: Prometone = 4.4 min

Atrazine = 4.7 min

Simazine = 4.9 min

Linearity checked: 0.2 ng - 20 ng

For Diuron confirmation:

Varian 6000 with TSD

Column: HP-1 (100% dimethyl polysiloxane) 10 m x 0.53 mm x 1.33 μ m

Carrier gas: Helium; Flow rate: 20 mL/min

Injector: 210°C

Detector: 250°C

Temperature program: Initial temp: 170°C held for 1 min

Rate: 10°C/min

Final temp: 220°C held for 1 min

Sample injected: 2 μ l

Retention time: Diuron = 3.2 min

Linearity checked: 0.2 ng - 20 ng

CONFIRMATION ANALYSIS:

For Bromacil confirmation:

Varian 6000 GC WITH TSD

Column: DB-1301 (6% cyanopropylphenyl & 94% methyl) 30 m x 0.55 mm
x 1.0 um

Carrier gas: Helium, flow rate: 20 mL/min

Injector: 220°C

Detector: 300°C

Temperature: 190°C isothermal

Injection volume: 2 uL

Retention time: Bromacil = 4.2 min

Linearity checked: 0.2 ng - 20 ng

The following results were obtained by the above method:

Sandy Soil

Chemical	Spike level (ppm)	Number of analyses (n)	Mean % Recovery	Standard Deviation* (±)
Atrazine	0.5	5	87.2	9.86
Prometone	0.5	5	86.0	10.00
Simazine	0.5	5	86.8	8.79
Bromacil	0.5	5	94.8	9.01
Diuron	0.5	5	83.2	6.42
Atrazine	2.0	5	78.6	5.76
Prometone	2.0	5	76.4	5.76
Simazine	2.0	5	78.9	5.72
Bromacil	2.0	5	85.5	3.23
Diuron	2.0	5	73.4	3.40
Atrazine	10.0	5	75.0	6.57
Prometone	10.0	5	89.2	6.56
Simazine	10.0	5	71.9	6.97
Bromacil	10.0	5	83.2	4.97
Diuron	10.0	5	87.8	4.37
Atrazine	40.0	5	75.8	6.74
Prometone	40.0	5	77.8	5.16
Simazine	40.0	5	66.4	6.26
Bromacil	40.0	5	80.6	4.34
Diuron	40.0	5	90.4	3.41

*Standard deviation of the spike recoveries.

Clay Soil

Chemical	Spike level (ppm)	Number of analyses (n)	Mean % Recovery	Standard Deviation (±)
Atrazine	0.5	5	68.4	2.97
Prometone	0.5	5	79.2	3.35
Simazine	0.5	5	68.0	3.16
Bromacil	0.5	5	72.4	3.58
Diuron	0.5	5	87.6	9.32
Atrazine	2.0	5	69.7	2.46
Prometone	2.0	5	73.5	3.00
Simazine	2.0	5	69.0	1.41
Bromacil	2.0	5	80.3	3.37
Diuron	2.0	5	92.4	5.02
Atrazine	10.0	5	90.6	2.67
Prometone	10.0	5	97.8	2.81
Simazine	10.0	5	85.7	4.03
Bromacil	10.0	5	80.3	2.45
Diuron	10.0	5	89.7	7.00
Atrazine	40.0	5	88.8	12.23
Prometone	40.0	5	87.4	8.60
Simazine	40.0	5	85.7	12.97
Bromacil	40.0	5	75.5	3.65
Diuron	40.0	5	89.8	4.24

CALCULATIONS:

$$\text{ppm} = \frac{(\text{peak height sample})(\text{ng/}\mu\text{L std})(\mu\text{L injected std})(\text{final volume (3 mL)})}{(\text{peak height std})(\mu\text{L injected sample})(\text{sample weight (5g)})}$$

DISCUSSION:

The minimum detection limit (MDL) for Bromacil by this method was 0.1 ppm and 0.05 ppm for Diuron, Atrazine, Simazine and Prometone. We can lower the MDL by increasing the sample size and lowering the final volume. However, this was not required for this project.

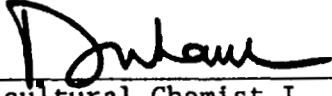
Several solvents such as hexane, acetone, methanol, and ethyl acetate were used in herbicide recovery studies. Because of each herbicide's different solubility, we found that no one solvent would give good recoveries for all chemicals. A mixture of hexane:acetone (60:40) was chosen since it gave relatively good recoveries for all analytes.

The hexane wash in step # 8 is necessary since it eliminates non polar compounds from the soil.

REFERENCE:

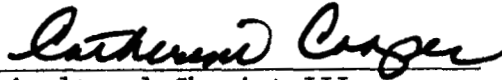
Duc Tran, Residue Analysis Of Atrazine in soil. 1989.

WRITTEN BY: Duc Tran



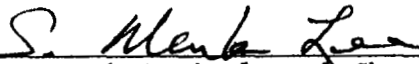
TITLE: Agricultural Chemist I

REVIEWED BY: Catherine Cooper



TITLE: Agricultural Chemist III

APPROVED BY: S. Mark Lee



TITLE: Research Agricultural Chemist

APPROVED BY: Terry Jackson



TITLE: Quality Assurance Officer

APPENDIX 2.

**MOISTURE CONTENT AND BULK DENSITY
IN SOIL CORE SAMPLES COLLECTED FROM THE PELTIER AND MANTECA BASINS**

Appendix 2. Moisture content and bulk density in soil core samples collected from the Peltier and Manteca basins.

Peltier basin, sampling site #13, 4/27/92

Depth (inches)	% Moisture	Bulk Density
0-6	12.5	1.75
6-12	9.2	1.82
12-18	10.1	1.63
18-24	9.2	1.63
24-30	10.5	1.28
30-36	9.1	1.17
36-42	7.4	1.05
42-48	11.0	1.22
48-54	5.5	1.15
54-60	7.8	1.18
60-72	9.9	1.22
72-84	16.7	0.92
84-96	12.3	0.95
96-108	14.4	1.68
108-120	11.1	1.73

Manteca SE basin, sampling site #20, 4/27/92

Depth (inches)	% Moisture	Bulk Density
0-6	8.8	1.38
6-12	9.2	1.27
12-24	9.2	1.32
24-36	16.8	1.65
36-48	18.2	1.50

Manteca NW basin, sampling site #18, 4/27/92

Depth (inches)	% Moisture	Bulk Density
0-6	17.4	1.57
6-12	19.6	1.50
12-18	22.6	1.37
18-24	24.7	1.22
24-30	20.0	1.00

APPENDIX 3.

**SOIL TEXTURE, ORGANIC CARBON, AND pH
IN SOIL CORE SAMPLES COLLECTED FROM THE PELTIER AND MANTECA BASINS**

Appendix 3. Soil texture, organic carbon, and pH in soil core samples collected from the Peltier and Manteca basins.

Depth (inches)	% of Sample >2.00mm	% Sand 0.05mm- 2.00mm	% Silt 0.002mm- 0.05mm	% Clay <0.002mm	pH	% Organic Carbon
North side of Peltier basin, sampling site #13						
0-6	2.4	60.0	23.0	17.0	7.1	0.2
6-12	1.3	70.0	16.0	14.0	7.0	0.1
12-24	2.8	74.0	12.0	14.0	6.8	0.1
24-36	2.8	78.0	7.0	15.0	6.7	0.0
36-48	4.9	81.0	7.0	12.0	6.9	0.0
48-60	3.8	86.0	6.0	8.0	7.0	0.0
60-72	1.0	62.0	28.0	10.0	7.1	0.0
72-84	0.7	64.0	26.0	10.0	7.1	0.0
84-96	0.5	66.0	21.0	13.0	7.1	0.0
96-108	0.2	60.0	29.0	11.0	7.1	0.0
108-120	0.1	64.0	26.0	10.0	7.2	0.0
South side of Peltier basin, sampling site #12						
0-6	0.0	62.0	26.0	12.0	7.5	0.1
6-12	0.0	48.0	40.0	12.0	7.7	0.0
12-24	0.1	50.0	38.0	12.0	7.6	0.0
24-36	0.0	52.0	34.0	14.0	7.3	0.0
36-48	0.0	60.0	30.0	10.0	7.4	0.0
48-60	0.1	74.0	19.0	7.0	7.2	0.0
60-72	0.2	74.0	20.0	6.0	7.1	0.0
72-84	0.3	83.0	11.0	6.0	6.9	0.0
84-96	0.1	96.0	2.0	2.0	6.8	0.0
96-108	0.0	84.0	14.0	2.0	6.9	0.0
108-120	0.1	86.0	10.0	4.0	6.7	0.0
Manteca SE basin, sampling site #20						
0-6	0.7	70.0	22.0	8.0	8.0	0.3
6-12	1.0	70.0	22.0	8.0	8.1	0.2
12-24	1.6	68.0	24.0	8.0	8.2	0.2
24-36	0.6	76.0	18.0	6.0	8.2	0.1
36-48	18.7	80.0	15.0	5.0	8.0	0.1
Manteca NW basin, sampling site #18						
0-6	0.3	52.0	34.0	14.0	7.8	0.8
6-12	1.1	44.0	50.0	6.0	8.1	0.1
12-24	0.2	30.0	48.0	22.0	8.0	0.1
24-36	0.3	62.0	25.0	13.0	8.2	0.1

Appendix 3. (continued)

Depth (inches)	% of Sample >2.00mm	% Sand 0.05mm- 2.00mm	% Silt 0.002mm- 0.05mm	% Clay <0.002mm	pH	% Organic Carbon
Highway 99 roadside, sampling site #14						
0-12	3.9	73.0	17.0	10.0	5.3	0.7
12-24	2.8	72.0	18.0	10.0	5.9	0.4
24-36	1.6	74.0	16.0	10.0	5.0	0.1
36-48	29.3	76.0	15.0	9.0	5.1	0.1
48-60	4.3	68.0	20.0	12.0	5.7	0.1
Highway 99 roadside, sampling site #15						
0-12	25.2	76.0	16.0	8.0	5.9	1.9
12-24	1.1	58.0	26.0	16.0	4.9	0.4
24-36	0.2	58.0	26.0	16.0	4.6	0.2
36-48	0.1	63.0	25.0	12.0	4.6	0.2
48-60	5.2	62.0	23.0	15.0	6.0	0.2
Highway 99 roadside, sampling site #16						
0-12	18.1	68.0	20.0	12.0	7.5	0.9
12-24	0.4	66.0	20.0	14.0	7.1	0.2
24-36	0.1	66.0	20.0	14.0	7.0	0.1
36-48	0.1	68.0	20.0	12.0	7.3	0.1
48-60	0.0	68.0	26.0	6.0	6.8	0.1

APPENDIX 4.

SAMPLE CHAIN-OF-CUSTODY FORM

30-031 (1/92)

Study #	Sample #	Sample Type	Subtype	Date			Time	Person	Collecting Location	Code	Segment Depth																												
				Mo	Day	Yr																																	
1	1	5									-	I N																											
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40
				Bromacil				Diuron				Simazine				Oryzalin				Oxyfluorfen				Lab Code															
																								4 3 2 3															

Partner:

Location:

Lab Results

(Save Extracts)

ppb (dry)

MDL

Companion Samples:

Bromacil

Diuron

Simazine

Oryzalin

Oxyfluorfen

Sample Dry Weight = _____ g

% Moisture = $\frac{(\text{Wet} - \text{Dry}) \times 100}{\text{Wet}}$ = _____

Extracted by:

Extraction Date:

Analyzed by:

Analysis Date:

Approved by:

Report Date:

Remarks

Key

Col 10-12 Sample Type

WAT = Water

SOI = Soil

Col 13 Subtype

I = Basin Inflow Water

S = Basin Stored Water

D = Basin Drywell Water

Col 26-27 Location

01 = Airport Way

02 = Union Road

03 = Peltier Road

Col 37

S = Spike

B = Blank Matrix

Task	Relinquished by	Received by	Date/Time
Container Prepared			
Collect/Transport			

Lab Name	Received for by lab	Date/Time	Logged in by	Date/Time	Lab #

Distribution: White to CDPR lab liaison, Yellow retained by lab, Pink to field files.